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APPLICATION NUMBER: 60/538,131

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Given Name (first and middle (if any))		Family Name or Surname		Residence (City and either State or Foreign Country)	
Arup K.		SenGupta		Bethlehem, PA	
Additional inventors are being named on the <u>2nd</u> separately numbered sheets attached hereto					
TITLE OF THE INVENTION (500 characters max)					
Hybrid Anion Exchangers (HAIX) for Selective Removal of Dissolved Arsenic Species and Other Ligands					
Direct all correspondence to: <b>CORRESPONDENCE ADDRESS</b>					
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ENCLOSED APPLICATION PARTS (check all that apply)					
<input checked="" type="checkbox"/>	Specification	Number of Pages	8	<input type="checkbox"/>	CD(s), Number
<input checked="" type="checkbox"/>	Drawing(s)	Number of Sheets	16	<input type="checkbox"/>	Other (specify)
<input checked="" type="checkbox"/>	Application Data Sheet. See 37 CFR 1.76				
METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT					
<input checked="" type="checkbox"/>	Applicant claims small entity status. See 37 CFR 1.27.				FILING FEE AMOUNT (\$)
<input checked="" type="checkbox"/>	A check or money order is enclosed to cover the filing fees.				\$80.00
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[Page 1 of 2]

Respectfully submitted,

SIGNATURE

TYPED OR PRINTED NAME George A. Smith, Jr.

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Date 1/21/01

REGISTRATION NO.

(If appropriate)

Docket Number:

24,442

SEN3USA

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Given Name (first and middle (if any))	Family or Surname	Residence (City and either State or Foreign Country)
Luis H.	Cumbal	Bethlehem, PA

(Page 2 of 2)

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**FEE TRANSMITTAL**  
**for FY 2004**

Effective 10/01/2003. Patent fees are subject to annual revision.

**Complete if Known**

Application Number	
Filing Date	
First Named Inventor	Arup K. SenGupta et al.
Examiner Name	
Art Unit	
Attorney Docket No.	SEN3USA

☒ Applicant claims small entity status. See 37 CFR 1.27

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**FEE CALCULATION****1. BASIC FILING FEE**

Large Entity Fee Code (\$)	Small Entity Fee Code (\$)	Fee Description	Fee Paid
1001 770	2001 385	Utility filing fee	
1002 340	2002 170	Design filing fee	
1003 530	2003 265	Plant filing fee	
1004 770	2004 385	Reissue filing fee	
1005 160	2005 80	Provisional filing fee	80.00
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**2. EXTRA CLAIM FEES FOR UTILITY AND REISSUE**

Total Claims	Extra Claims	Fee from below	Fee Paid
0	-20** =	X	0.00
Independent Claims	0	-3** =	0.00
Multiple Dependent			

Large Entity Fee Code (\$)	Small Entity Fee Code (\$)	Fee Description
1202 18	2202 9	Claims in excess of 20
1201 86	2201 43	Independent claims in excess of 3
1203 290	2203 145	Multiple dependent claim, if not paid
1204 86	2204 43	** Reissue Independent claims over original patent
1205 18	2205 9	** Reissue claims in excess of 20 and over original patent

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**FEE CALCULATION (continued)****3. ADDITIONAL FEES**

Large Entity Small Entity

Fee Code (\$)	Fee Code (\$)	Fee Description	Fee Paid
1051 130	2051 65	Surcharge - late filing fee or oath	
1052 50	2052 25	Surcharge - late provisional filing fee or cover sheet	
1053 130	1053 130	Non-English specification	
1812 2,520	1812 2,520	For filing a request for ex parte reexamination	
1804 920*	1804 920*	Requesting publication of SIR prior to Examiner action	
1805 1,840*	1805 1,840*	Requesting publication of SIR after Examiner action	
1251 110	2251 55	Extension for reply within first month	
1252 420	2252 210	Extension for reply within second month	
1253 950	2253 475	Extension for reply within third month	
1254 1,480	2254 740	Extension for reply within fourth month	
1255 2,010	2255 1,005	Extension for reply within fifth month	
1401 330	2401 165	Notice of Appeal	
1402 330	2402 165	Filing a brief in support of an appeal	
1403 290	2403 145	Request for oral hearing	
1451 1,510	1451 1,510	Petition to institute a public use proceeding	
1452 110	2452 55	Petition to revive - unavoidable	
1453 1,330	2453 665	Petition to revive - unintentional	
1501 1,330	2501 665	Utility issue fee (or reissue)	
1502 480	2502 240	Design issue fee	
1503 640	2503 320	Plant issue fee	
1460 130	1460 130	Petitions to the Commissioner	
1807 50	1807 50	Processing fee under 37 CFR 1.17(q)	
1806 180	1806 180	Submission of Information Disclosure Stmt	
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1801 770	2801 385	Request for Continued Examination (RCE)	
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Date 1/21/04

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## **Hybrid Anion Exchangers (HAIX) for Selective Removal of Dissolved Arsenic Species and Other Ligands**

**Inventors: Arup K. SenGupta and Luis Cumbal**

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### **Invention Synopsis**

Using a new chemical-thermal technique, polymeric anion exchangers have for the first time been used as the host materials in our laboratory for irreversibly dispersing Hydrated Fe(III) Oxides (HFO) within the spherical exchanger beads. Since the anion exchangers have positively charged quaternary ammonium functional groups, anionic ligands such as arsenates, chromates, oxalates, phosphates, phthalates can permeate in and out of the gel phase and are not subject to Donnan exclusion effect. Consequently, the anion exchanger supported HFO micro particles, referred to as Hybrid Anion Exchanger or HAIX, showed significantly greater arsenic or ligand removal capacities in comparison with cation exchanger supports, all other conditions remaining identical. Polymeric anion exchangers do not have any capacity for ferric ( $\text{Fe}^{3+}$ ) or ferrous ( $\text{Fe}^{2+}$ ) cations. Therefore, their uses as host materials for HFO for selective sorption of ligands have not been previously reported in the open literature.

Newly prepared HAIX materials are amenable to efficient regeneration with sodium hydroxide and sodium chloride, and can be reused for multiple cycles without significant loss in arsenic removal capacity. An extensive investigation pertaining to arsenic removal from contaminated groundwaters clearly shows that HAIX far outperforms the cation exchanger doped HFO particles studied earlier.

## Previous Studies: Limitations of Cation Exchangers as Host Materials

### Selected References:

1. US Patent No. 5,453,201. Water Treatment Process. J.E. Etzel and J. Kurek. Date: Sept. 26, 1995.
2. US Patent No. 3,984,313. Preferential removal of ammonia and phosphates. I.R. Higgins. Date: Oct. 5, 1976.
3. Arsenic removal using a polymeric/inorganic hybrid sorbent. M.J. DeMarco, A.K. SenGutpa and J.E. Greenleaf. *Water Research*, 37 (2003), 164-176.
4. Arsenate sorption by Fe(III)-doped alginate gels. J.M. Min and J. Hering. *Water Research*, 32 (1998), 1544-1552.
5. Removing selenium(IV) and arsenic(V) oxyanions with tailored chelating polymers. A. Ramana and A.K. SenGupta. *J. Environ. Eng. Div. ASCE*. 118 (1992), 755-775.
6. Surface structures and stability of arsenic on goethite surface: spectroscopic evidence for inner-sphere complexes. B.A. Manning, S.E. Fendorf and S.E. Goldberg. *Environ. Sci. Technol.*, 32 (1998), 2383-2388.

It is universally recognized that a fixed-bed sorption process is operationally simple, requires virtually no start-up time and is forgiving toward fluctuations in feed compositions. However, in order for the fixed-bed process to be viable and economically competitive, the sorbent: i) must exhibit high selectivity toward the target contaminant, ii) be durable and iii) amenable to efficient regeneration and reuse. Ideally, the removal of the target contaminant should not cause major changes in pH or in the composition of the influent water. In this regard, both amorphous and crystalline Hydrated Fe Oxide (HFO) show strong sorption affinity toward both As(III) and As(V) oxyacids and oxyanions through ligand exchange in the coordination spheres of structural Fe atoms. Recent investigations using extended X-ray absorption fine structure (EXAFS) spectroscopy confirmed that As(III) and As(V) species are selectively bound to the oxide surface through formation of inner sphere complexes. HFO particles also exhibit high sorption affinities toward phosphate, natural organic matters, selenite and other anionic ligands. **Figure 1** shows an illustration of the binding of various solutes onto hydrated

Fe(III) oxides or HFO. Note that commonly encountered competing ions, such as chloride or sulfate can be sorbed only through Coulombic interaction or formation of outer sphere complexes. Thus, they exhibit poor sorption affinity toward HFO particles. On the contrary, ligands such as arsenite, monovalent arsenate, divalent arsenate, phosphate etc. are sorbed strongly through Lewis acid-base interaction or formation of inner sphere complexes.

However, the traditional process of syntheses, although straightforward, produces only very fine submicron HFO particles which are unusable in fixed beds, permeable reactive barriers or any flow-through systems because of excessive pressure drops, poor mechanical strength and unacceptable durability. In order to overcome this problem, the strong-acid cation exchangers have previously been modified to dope/disperse HFO particles for removal of phosphates. Iron-loaded cation exchange resins and alginates have also been tried to remove selenium and arsenic oxyanions. Although cation-exchanger-loaded hydrated Fe(III) oxide (HFO) particles are capable of removing arsenates or phosphates, the removal capacities are reduced due to the following reason: the gel phase of the cation exchanger is negatively charged due to the presence of the sulfonic acid groups. That is why arsenates or As(V) oxyanions and phosphates are rejected due to Donnan co-ion exclusion effect and dispersed HFO particles in the gel phase are not accessible to dissolved anionic ligands for selective sorption. When we used macroporous cation exchangers as the host materials, arsenic removal capacity was not high but quite reasonable and in the order of 750  $\mu\text{g As/g}$  of sorbent. However, when gel-type cation exchanger was used for dispersing HFO particles, the resulting material was ineffective altogether. **Figure 2** shows a column run effluent history where a gel-type cation exchanger has been loaded with eight percent HFO present as Fe. Note that the arsenic breakthrough took place almost immediately i.e., the material had practically no arsenic removal capacity. This observation provided a significant clue that HFO particles when encapsulated within cation exchange sites are not accessible to arsenates or other anionic ligands for selective sorption as illustrated in **Figure 3**. At the same time, dispersing HFO particles within a cation exchanger material is a relatively straight forward process and the same has been carried out previously by several researchers/inventors.



### Underlying Hypothesis

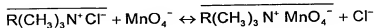
Unlike cation exchangers, anion exchangers have fixed positively charged functional groups. Thus, anionic ligands can easily permeate in and out of the gel phase without encountering Donnan co-ion exclusion effect. We hypothesized that if HFO particles could by some means be dispersed within a polymeric anion exchanger bead, arsenic or ligand removal capacity will significantly increase. On the other hand, forming hydrated Fe(III) oxides within an anion exchange resin poses a major challenge due to positively charged quaternary ammonium functional groups. No previous report of dispersing HFO particles within a polymeric anion exchanger is recorded in the open literature.

### Stepwise Protocol for Preparation of Anion Exchanger Supported HFO Particles

Since  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$  are cations, they cannot be loaded onto the anion exchange resins. Thus, the technique used earlier for dispersing HFO particles within cation exchanger beads is not applicable when anion exchanger beads serve as the host materials. After having gone through several trial and errors, the following step-wise protocol was successfully developed and refined for dispersing HFO within polymeric anion exchange resins:

#### Step 1. Loading of Permanganate Anion ( $\text{MnO}_4^-$ )

Permanganate anion was loaded onto the anion exchanger (Purolite A400) by passing permanganate solution (500 mg/L  $\text{KMnO}_4$ ) through the bed.

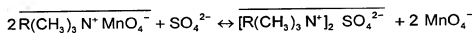


$\text{R}(\text{CH}_3)_3\text{N}^+\text{Cl}^-$  = Anion exchange resin with quaternary ammonium functional group in chloride form. Anion exchange resins from other companies besides Purolite Inc. may also be used. Particle sizes of anion exchange resins used in our laboratory varied from 300  $\mu\text{m}$  to 1000  $\mu\text{m}$ .

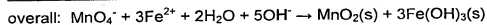
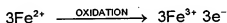
## Step II. Concurrent Permanganate Desorption, Fe(II) Oxidation and HFO Formation Within Anion Exchanger

During this step, permanganate loaded anion exchanger was brought in contact with 5% ferrous sulfate solution. Desorption of  $\text{MnO}_4^-$  by sulfate, reduction of  $\text{MnO}_4^-$  to  $\text{MnO}_2(\text{s})$  and oxidation of  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$  and finally, precipitation of  $\text{Fe}(\text{OH})_3(\text{s})$  within the anion exchanger beads took place in accordance with the following:

$\text{MnO}_4^-$  Desorption:



Fe(II) Oxidation and Formation of Ferric Hydroxide:



overbar and (s) denote solid phase.

## Step III. Acetone Wash and Drying

Anion exchanger beads from Step II was washed with acetone and dried in a furnace at 35°C for eight hours.

**Figure 4** depicts the major steps of the process. Step I and Step II can be repeated for the second time to achieve greater Fe(III) loading. During Step III, the use of acetone lowered the dielectric constant of water and enhanced the agglomeration of HFO

submicron particles through suppression of surface charges. HFO agglomerates were irreversibly encapsulated within the spherical anion exchanger beads; turbulence and mechanical stirring did not result in any noticeable loss of HFO particles. Instead of permanganate, other oxidizing anions, namely, hypochlorite or  $\text{OCl}^-$ , may also be used.

Both gel and macroporous anion exchanger beads (Purolite A400 and A500 P) were used in the study. Mass loading of HFO onto the anion exchanger varied from 10-15% as Fe; the manganese content was less than 1% by mass as Mn. **Figure 5A** shows Hybrid Anion Exchanger (HAIX) particles. **Figure 5B** shows the scanning electron microphotograph (SEM) of a sliced parent polymer bead; the presence of macropores can be readily observed. **Figure 5C** shows a sliced HAIX particle containing HFO nanoparticles. While the polymeric anion exchanger beads offer excellent hydraulic properties and durability during fixed-bed column runs, the dispersed HFO microparticles serve as active sorbents for target ligands.

## Performance Evaluation of HAIX

### Experimental Protocol:

A series of fixed-bed experiments were carried out in our laboratory to validate excellent As(III) and As(V) removal capacities of HAIX. The experimental set-up is illustrated in **Figure 6**. Tests were also conducted to validate HAIX's ability to remove chromate, phosphate and natural organic matter. The following results are noteworthy:

#### 1. As(III) Removal

**Figure 7A - C** provides As(III) effluent histories for three separate column runs under nearly identical conditions using: A) parent Anion Exchanger Purolite A-500P B) HFO loaded cation exchanger reported earlier and C) Hybrid Anion Exchanger or HAIX.

The parent anion exchanger was unable to remove any As(III) (**Fig. 7A**) but the HFO loaded cation exchanger removed As(III) over 2000 bed volumes corresponding to a breakthrough concentration of 10  $\mu\text{g/L}$  (**Figure 7B**). However, HAIX or HFO loaded

anion exchanger treated nearly 12,000 bed volumes of contaminated feed water before the arsenic concentration reached 10 µg/L (**Figure 7C**).

## 2. As(V) Removal

**Figures 8 and 9** show the comparison of arsenic effluent histories between two column runs under identical conditions, one with cation exchanger loaded HFO and the other with HAIX. Total HFO loading for both materials was comparable and in the vicinity of 10% by mass as Fe. Note that HAIX offered much greater arsenic breakthrough capacity.

## 3. Regeneration

Arsenic loaded HAIX column was very efficiently regenerated using 3% NaCl + 2% NaOH with an empty bed contact time of five minutes. In less than 10 bed volumes over 90% of arsenic was desorbed (**Figure 10**).

## 4. Simultaneous Removal of Arsenate and Chromate

HAIX column was fed with a representative synthetic feed water containing both trace concentrations of arsenic and chromium (VI). Note that the HAIX column was very effective in removing both arsenic(V) and chromium(VI) simultaneously up to nearly 2000 bed volumes (**Figure 11**). Upon exhaustion, the column was regenerated using 2% NaOH and 3% NaCl. **Figure 12** shows that the desorption of arsenate and chromate was very efficient.

## 5. Removal of Phosphate

HAIX column was fed with a feed solution containing phosphate. **Figure 13** shows the effluent history of P confirming HAIX's ability to selectively remove phosphate in the presence of other competing anions, namely, chloride and sulfate.

## 6. Reuse for Multiple Cycles

One HAIX column was run successively for three cycles. After each cycle, the column was regenerated with 2% NaOH and 3% NaCl. **Figure 14** shows the effluent arsenic histories for two successive column runs using groundwater collected from a

contaminated site in Ontario, Canada. Note that arsenic effluent histories remained essentially the same. These results provide evidence that HAIX can be regenerated and reused for multiple cycles without any significant loss in arsenic removal capacity.

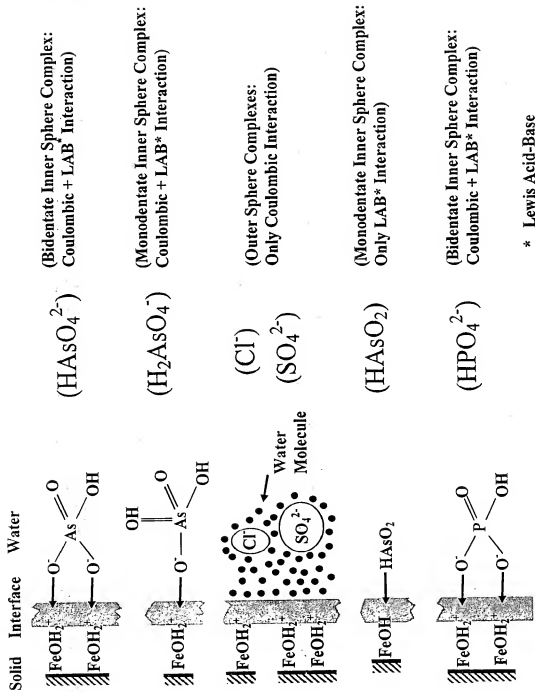


Figure 1 An illustration of the binding of various solutes onto hydrated Fe(III) oxides (HFO)

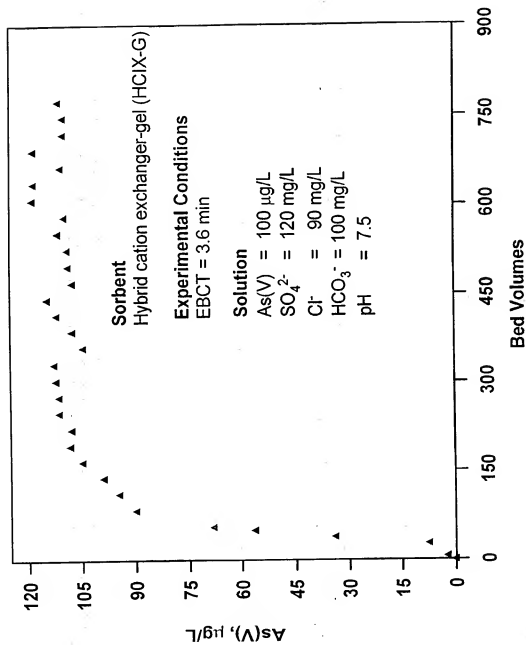
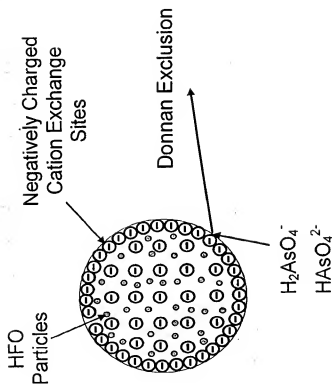


Figure 2 Effluent history for arsenic during a column run using the hybrid cation exchanger-gel (HCIX-G)

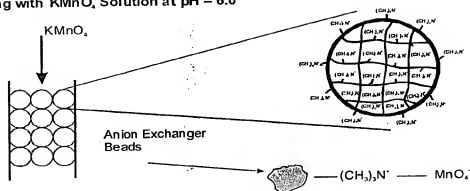


Remark: HFO inside the gel phase are not available for selective sorption of arsenate

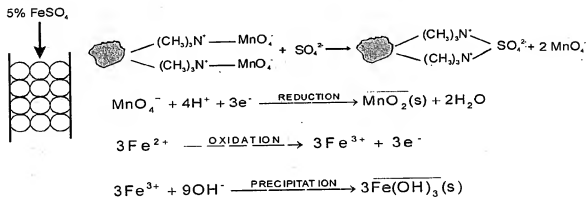
**Figure 3** An illustration of the mechanism explaining why HFO doped cation exchange resins are unable to offer significant arsenic capacity



**Step 1. Loading with  $\text{KMnO}_4$  Solution at  $\text{pH} = 6.0$**



**Step 2. Desorption and simultaneous redox/precipitation in the gel phase and pores at  $\text{pH} < 4.0$**



**Step 3. Acetone Wash and Drying**

**Figure 4** Illustration of the three-step protocol for preparing HAIX sorbents



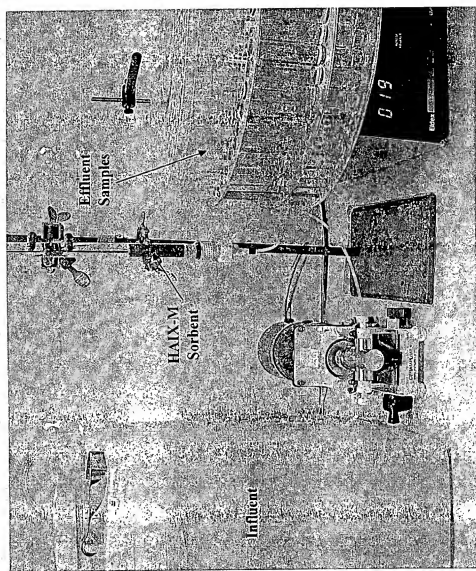
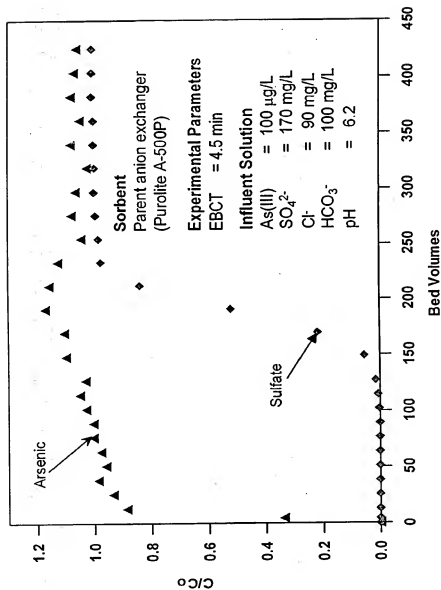
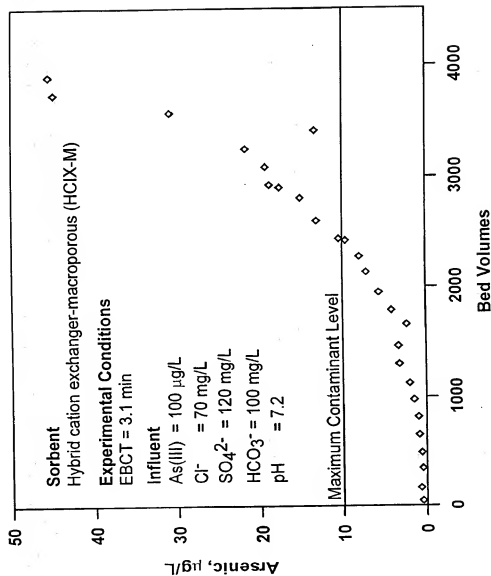


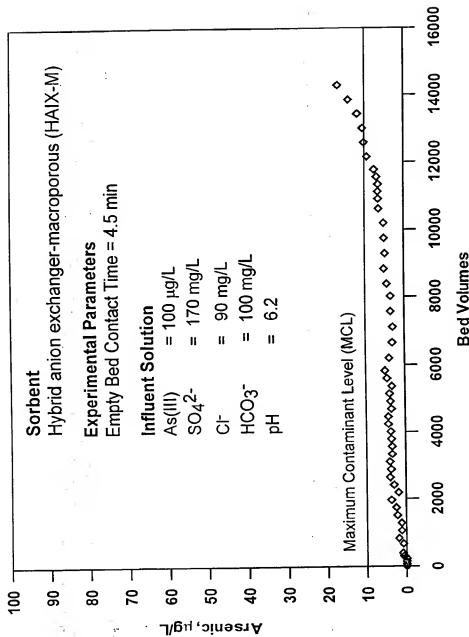
Figure 6 The laboratory set-up for fixed-bed column experiments.



C/Co = Fraction of the influent concentration present at the exit of the column

**Figure 7(A)** Effluent histories of sulfate and arsenic during a column run with parent anion exchanger (A-500P) in chloride form. Arsenic breaks through from the column immediately and long before sulfate. The anion exchange resin does not have any As(III) removal capacity.





**Figure 7(c)** Effluent history of arsenic during a column run with a hybrid anion exchanger-macroporous (HAIX-M).  
Remarks: HAIX-M offers six-fold greater As(III) removal capacity than HCIX-M

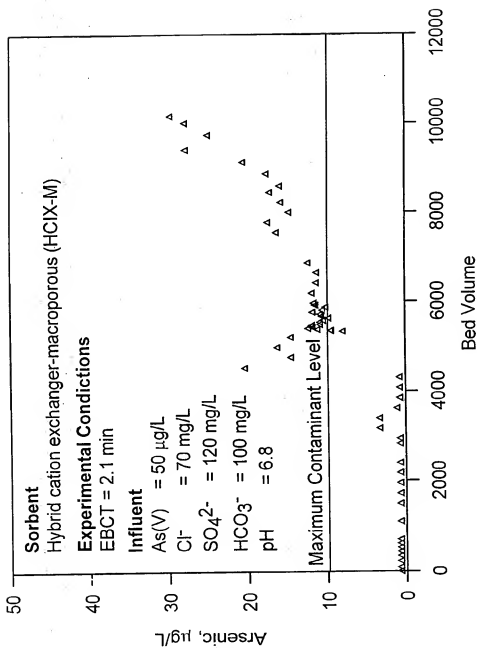
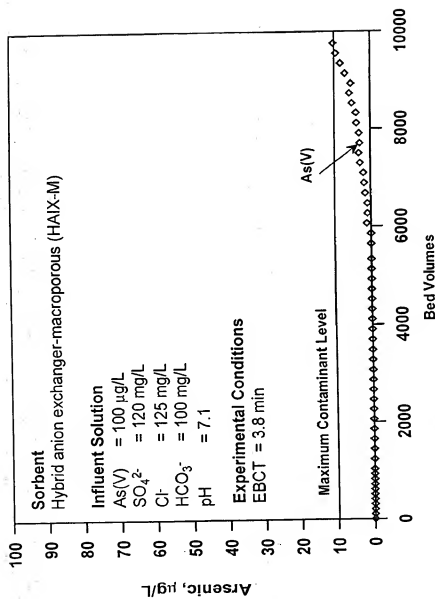


Figure 8 Effluent history of As(V) during a column run with a hybrid cation exchanger-macroporous (HCIX-M).



**Figure 9** Effluent history of arsenic during a column run with the hybrid anion exchanger-macroporous (HAIX-M).  
Remarks: HAIX-M offers significantly greater arsenic removal capacity



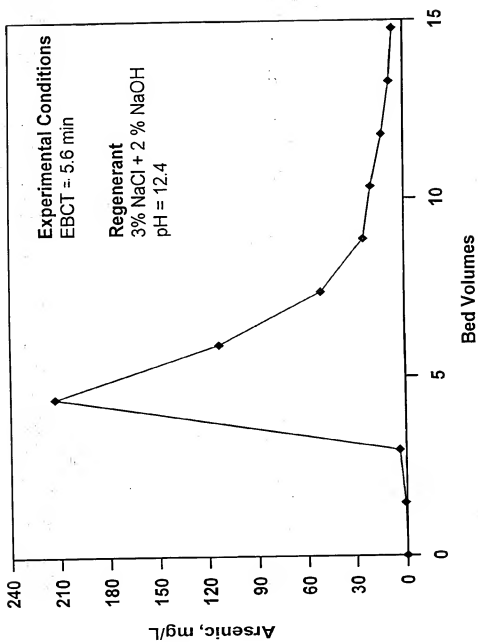
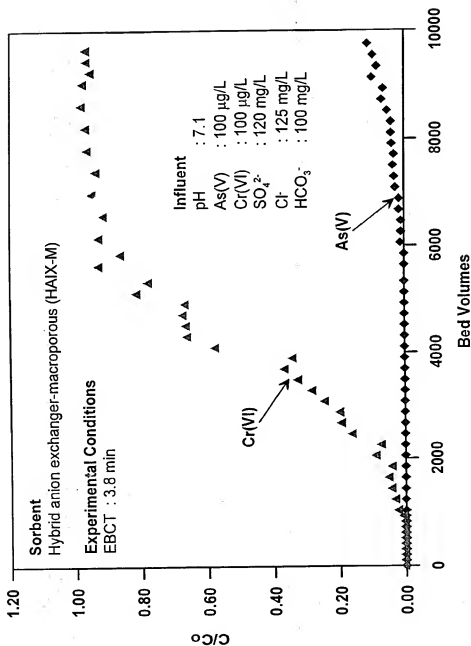


Figure 10 Arsenic concentration profile during regeneration of HAIX-M.



**Figure 11** Effluent histories of As(V) and Cr(VI) during a column run with the hybrid anion exchanger-macroporous (HAIX-M).

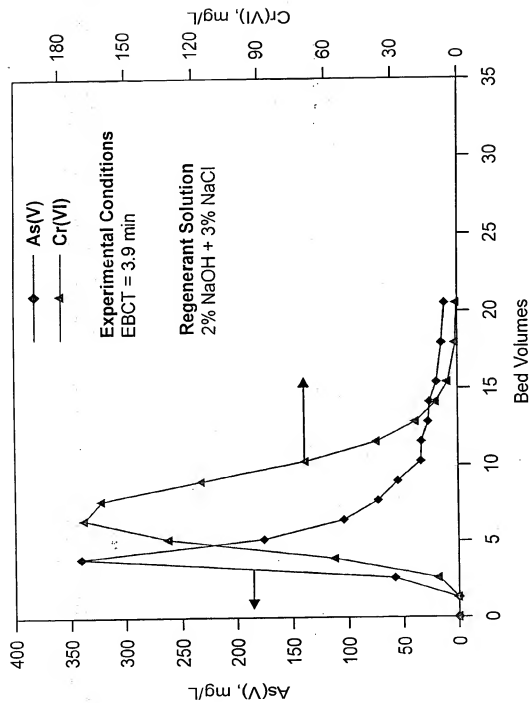


Figure 12 Concentration profiles of arsenic and chromium during regeneration of HAIX-M.

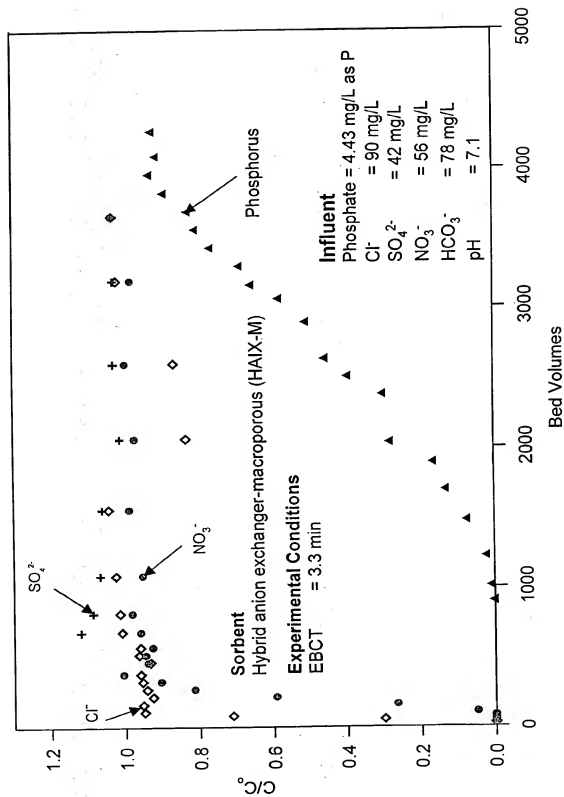
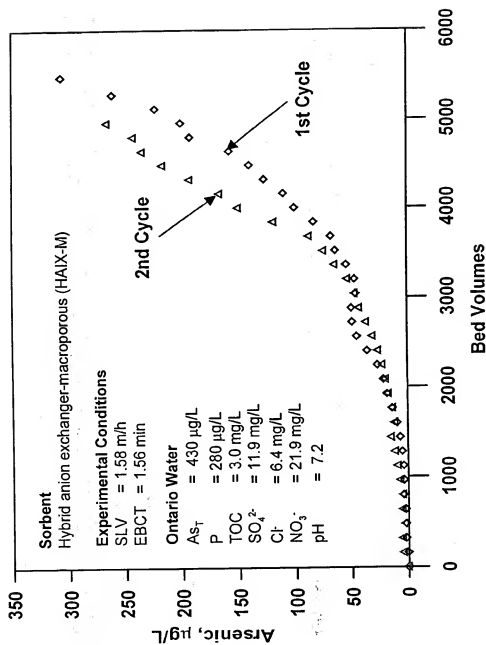


Figure 13 Breakthrough profiles for phosphorus and other anions during a column run using HAIX-M.



**Figure 14** Effluent histories for arsenic during two consecutive column runs using HAIX-M with the same contaminated groundwater feed.

## APPLICATION DATA SHEET

### Application Information

Application Type::	Provisional
Subject Matter::	Utility
Suggested Classification::	
Suggested Group Art Unit::	
CD-ROM or CD-R?::	None
Number of CD disks::	
Number of Copies of CDs::	
Sequence Submission?::	
Computer Readable Form (CRF)?::	
Number of Copies of CRF::	
Title::	HYBRID ANION EXCHANGERS (HAIX) FOR SELECTIVE REMOVAL OF DISSOLVED ARSENIC SPECIES AND OTHER LIGANDS
Attorney Docket Number::	SEN3USA
Request for Early Publication::	No
Request for non-Publication::	No
Suggested Drawing Figure::	
Total Drawing Sheets::	16
Small Entity::	Yes
Latin name::	
Variety denomination name::	
Petition Included?::	No
Petition Type::	
Licensed US Govt. Agency::	
Contract or Grant Numbers::	
Secrecy Order in Parent Application::	No

## **Applicant Information**

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State or Province of Mailing Address::	PA
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Postal or Zip Code of Mailing Address::	18017

### **Applicant Information**

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Telefacsimile:: 215-540-5818  
E-Mail Address:: gasmith@howsonandhowson.com

**Representative Information**

Representative Customer Number::	00270	
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**Domestic Priority Information**

Application::	Continuity Type::	Parent Application::	Parent Filing Date::
This Application			

**Foreign Priority Information**

Country::	Application Number::	Filing Date::	Priority Claimed::

**Assignee Information**

Assignee Name::

Street of Mailing Address::

City of Mailing Address::

State or Province of Mailing Address::

Country of Mailing Address::

Postal or Zip Code of Mailing Address::